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Experimental Study of X-ray Diffraction under Specular Reflection Conditions

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Abstract

Results of the experimental study of X-ray diffraction under specular reflection conditions are presented. The experimental arrangement which permits the measurement of the intensity of a specularly reflected diffracted wave with respect to its exit angle to the crystal surface is described. Experimental confirmation of the theory [Afanas'ev & Melkonyan (1983). *Acta Cryst.* **A39**, 207–210] has been obtained for silicon crystals. The angular distributions of the specularly reflected and diffuse intensities have also been studied. The experiments showed the diffuse

scattering to be primarily scattering at the back edge of the sample.

The diffraction under specular reflection conditions occurs when an X-ray beam is directed into a crystal at a small glancing angle, φ , of incidence comparable with the critical angle of specular reflection and, simultaneously, if the conditions of Laue case diffraction for the planes normal to the surface are met.

As was shown earlier (Marra, Eisenberger & Cho, 1979; Afanas'ev & Melkonyan, 1983), two specularly

reflected waves leave the crystal through the entrance surface in this case. The former wave satisfies the specular reflection condition, making an angle φ with the surface. The latter one being the specular component of the diffracted wave, makes an angle $2\theta_B$ with the specularly reflected wave. The value of the exit angle φ_h made by this wave with the surface depends on how exactly the Bragg condition is met. In particular, the following relation occurs:

$$\alpha = \varphi^2 - \varphi_h^2, \quad (1)$$

$$\alpha = -2 \sin(2\theta_B) \delta\theta, \quad (2)$$

the parameter determining the deviation from the Bragg condition.

Analytical expressions defining the specularly reflected diffracted intensity dependence on φ and α have been obtained by Afanas'ev & Melkonyan (1983). It has also been shown that at certain values of the glancing angle φ the reflection-coefficient maximum is close to unity.

The difficulty of experimental realization of diffraction under specular reflection conditions is connected with the necessity of X-ray beam collimation in two mutually perpendicular planes: through the glancing angle φ (of the order of minutes of arc) and through the angular deviation $\delta\theta$ from the exact Bragg angle (of the order of seconds of arc).

The use of (1) enables one to avoid the problem and to give up the collimation through the angle $\delta\theta$ (i.e. on α). Then, instead of a single specularly reflected wave some set of the waves will exit resulting in an intensity distribution depending on the angle φ_h . Recording this distribution as a function of the angle φ_h , i.e. introducing collimation through the exit angle φ_h , for example, with an accuracy of $30''$, one has an accuracy in $\delta\theta$ of

$$\delta\theta = \frac{\varphi \delta\varphi_h}{\sin(2\theta_B)}.$$

To solve this problem experimentally a new experimental scheme was proposed by Golovin & Imamov (1983). This scheme allows one to measure the dependence of specularly reflected intensity on φ in the integral mode, i.e. with integral registration through all exit angles φ_h , which is the simplest to perform.

The results of such measurements with silicon single crystals were presented (Golovin & Imamov, 1983). Integral curves can be used for obtaining preliminary information on the surface layers. However, to obtain detailed information on these layers measurements in differential mode with a narrow interval at the exit angles are required. The angular dependences of intensity measurements in the differential mode for a perfect silicon single crystal are presented below.

A sketch of the experimental set-up is shown in Fig. 1. An X-ray beam from a 1.2 kW X-ray source

was directed at a monochromator crystal at the Bragg angle [silicon (220)-symmetrical reflection in the Bragg case for Cu $K\alpha$ radiation was used]. The beam reflected by the monochromator crystal was incident on the sample at a small glancing angle φ and diffracted on the (220) planes normal to the entrance surface.

There was a slit (see Fig. 1, no. 3) between the monochromator crystal and the sample to distinguish a $K\alpha_1$ line from the total X-ray spectrum. The slit size defined the width of the incident-beam front (20–100 μm). The beam divergence in the horizontal plane, in our experiment about $6''$, was defined by the angular half-width of the monochromator rocking curve. In the vertical plane collimation was not provided and the divergence of the X-ray beam was limited by the design of the spectrometer. In the experimental set-up the vertical divergence was about 1° .

The angular dependences were measured by a goniometer based on a triple-crystal spectrometer (TRS).

Counters 1 and 2 were set on bars rotating about the goniometer axis.

The first counter was supplied with a slit for registering diffracted waves with different exit angles φ_h from the crystal surface. The slit had an alterable window which was used to distinguish a rather narrow interval from the whole band of φ_h angles. Since the axis around which the angle was to be measured made an angle $2\theta_B$ with the goniometer axis, the turn of counter 1 around the goniometer axis by an angle

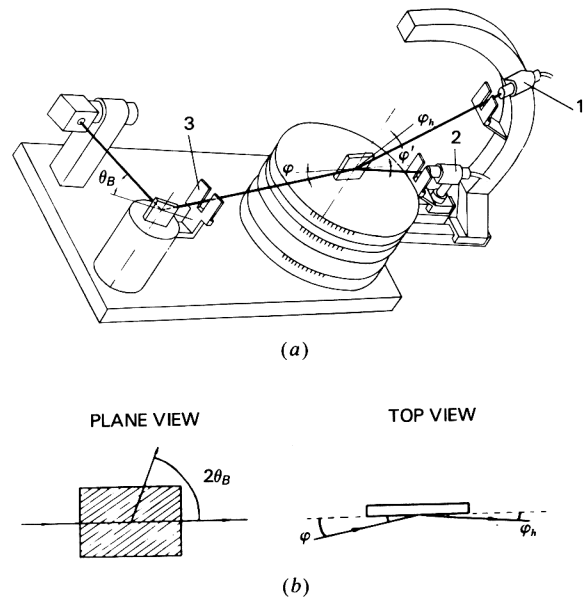


Fig. 1. X-ray optics scheme of the experimental set-up. General view of the experimental arrangement: 1 counter for registration of specularly reflected diffracted intensity; 2 counter for registration of specularly reflected and forward-beam intensities; 3 slit. (b) Scheme of beam trajectory.

$\delta\varphi_1$ corresponded to a change of φ_h of

$$\delta\varphi_h = \delta\varphi_1 \cos 2\theta_B.$$

In the experiment only part of the beam was incident onto the sample, the remaining (forward) beam passing it by and forming a reference signal for counter 2. Counter 2 with a removable slit $50\ \mu\text{m}$ in width was used for investigation of the X-ray intensity angular spread in the direction of the forward beam and specularly reflected beam. The angular position φ' of detector 2 was measured around the goniometer axis from the forward-beam direction, and the direction of the specular reflected beam in this reference frame corresponded to $\varphi' = 2\varphi$.

A multichannel analyzer NTA-1024 working in a multichannel mode was attached to the system as a recording device. The duration of pulses accumulation in each channel was 2 s.

The experiments were carried out with a perfect silicon single crystal with (100) surface orientation after standard mechanical and chemical surface treatments. The deviation of surface orientation from (100) was less than $\pm 15'$. Crystal diameter was 20 mm, thickness 1 mm.

Typical diffractograms measured with detector 1 and showing dependences of specular reflected

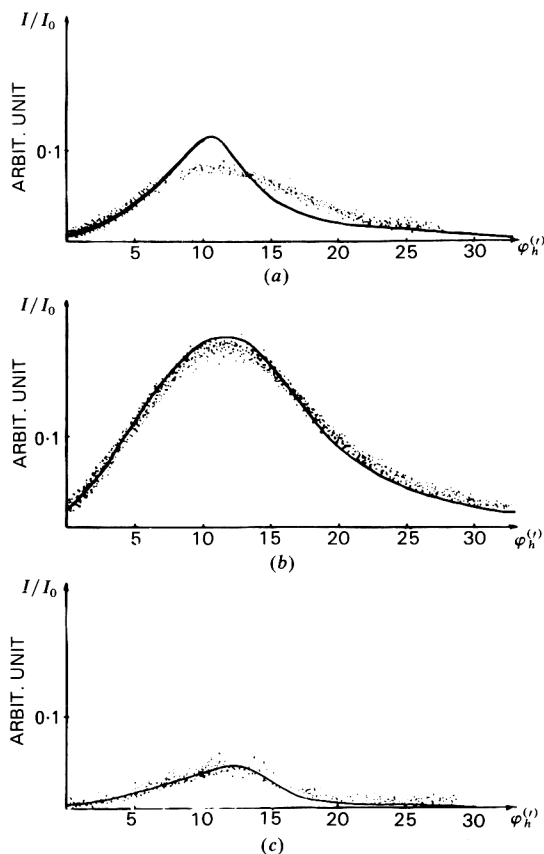


Fig. 2. Typical diffractograms for silicon samples and different angles φ of incidence. (a) $\varphi = 4'$, (b) $\varphi = 12'$, (c) $\varphi = 26'$.

diffracted intensity on φ_h for different glancing angles of incidence φ (4, 12 and $26'$) are presented in Fig. 2. The resolution at φ_h was not worse than $3'$ because the slit size at counter 1 was $50\ \mu\text{m}$. Fig. 2 shows the calculated curves which were obtained on the basis of the theory (Afanas'ev & Melkonyan, 1983), averaging through the angle φ_h over $3'$ and with an allowance for a relative contribution of polarizations $I_\sigma/I_\pi = \cos 2\theta_B = 0.7$.

$$I(\varphi, \varphi_h) \approx \sum_{j=\delta, \pi} c_j \int_{\varphi_h - 0.5\Delta\varphi_h}^{\varphi_h + 0.5\Delta\varphi_h} P_{hj}^s(\varphi, \varphi_h)(2\varphi_h) d\varphi_h$$

where $c_\pi = 1/(1 + \cos 2\theta_B)$, $c_\delta = c_\pi \cos 2\theta_B$, $\Delta\varphi_h = 3'$, $(2\varphi_h)$ being the factor due to (1). The value $P_{hj}^s(\varphi, \varphi_h)$ is the reflection coefficient calculated by Afanas'ev & Melkonyan (1983). Theoretical and experimental curves show satisfactory agreement in shape, relationship of intensities and the intensity maxima angular position.

However, some differences are evident. First of all, there is disagreement in theoretical and experimental half-width values. It can be especially well seen in the spectrum corresponding to the glancing angle $\varphi = 4'$; at this glancing angle the X-ray penetration depth is small, less than $100\ \text{\AA}$, and there are many imperfections in subsurface layers of real crystals. Besides, a possible disorientation of the surface from (100) and the influence of surface unevenness were not taken into account.

The angular distributions of the forward-beam intensity both for the case when the sample is mounted on the goniometer and makes different angles with the beam and for the case without the sample are presented in Fig. 3. Distributions of specularly reflected intensity are also presented. One can see

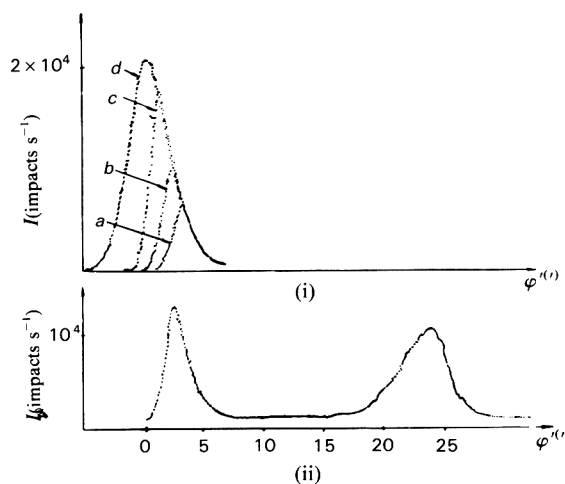


Fig. 3. (i) Distribution of the forward-beam intensity with the crystal making different angles with the beam: (a) $\varphi = 26'$, (b) $\varphi = 12'$, (c) $\varphi = 0'$, (d) without the crystal. (ii) Distributions of specularly reflected and diffuse intensities for $\varphi = 12'$.

that the specular intensity maximum corresponds to the angle φ' equal to 2φ . Besides the specularly reflected intensity diffuse scattering is observed, which might be caused by the surface unevenness and by the back edge of the sample. It is registered most clearly at the angle $\varphi > 14'$, i.e. in the case when the specularly reflected intensity may be neglected (see

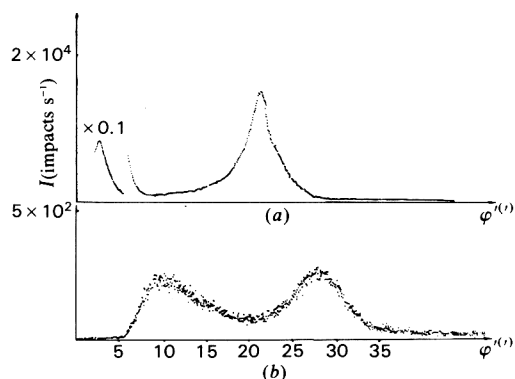


Fig. 4. Distribution of the diffuse intensity for $\varphi = 16'$. (a) The crystal covers half of the beam (the forward wave can be seen); (b) the crystal covers the whole beam (no forward beam).

Fig. 4a). The experiments showed a weakening of the diffuse intensity when only part of the incident beam reached the crystal back edge (see Fig. 4b). This fact showed that the main contribution to the diffuse scattering was due to the scattering at the sample back edge.

Thus, the intensity measurement of the specularly reflected diffracted wave with its exit-angle change has been experimentally realized and the main statements of the theory (Afanas'ev & Melkonyan, 1983) have been confirmed.

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